Synthesis of Crude Palm Oil-Based Polyol Ester as Biolubricant

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Abstract

The decline in crude oil reserves as a raw material for manufacturing lubricants and the need to reduce pollution from mineral lubricants has encouraged waste and pollution reduction technologies to reduce environmental problems. One realization is an alternative lubricant called bio lubricant. Biolubricant is considered rapidly biodegradable, non-toxic, and thought to pose less or no risk to the environment and its users than petroleum-based lubricants. This study aimed to produce polyol esters as bio-lubricants through the acetylation reaction of polyols from crude palm oil and investigate the resulting polyol esters' physical and chemical properties. From the study results, the kinematic viscosity of 40 °C in the resulting polyol ester was 46.2339 cSt, while the kinematic viscosity at 100 °C was 8.7417 cSt. The viscosity index and flash point of polyol ester were 144.0818 and 282.3 °C. The highest conversion of 38.41% polyol ester production was achieved at 80 °C for 35 minutes using a 3% wt/wt catalyst. This study shows that palm oil esters have great potential to obtain lubricants with high biodegradability and properties.

Keywords: acetylation, bio lubricant, bentonite, crude palm oil, polyol ester

INTRODUCTION

Currently, around 40% of lubricants worldwide are dominated by petroleum derivative products, so they harm the environment (do Valle et al., 2018). In recent years, attention to pollution and environmental health has attracted attention regarding the emphasis on switching from non-biodegradable lubricants to environmentally friendly products derived from growing biomass. Using biodegradable lubricants has reduced environmental pollution (Kamalakar et al., 2015).

Research on bio-based materials for industrial and automotive applications has accelerated recently (Heikal et al., 2017). The raw materials commonly used to develop lubricants

are derived from vegetable oils. From a technical point of view, 90% of lubricants that can decompose naturally use vegetable oil products and modified vegetable oil esters as essential ingredients (Zhang et al., 2020). Vegetable oils are triglycerides, glycerol esters, and three straight-chain fatty acids. Fatty acid chain length usually ranges from C12-C24. These three fatty acids vary between feedstocks and play an essential role in determining the properties of the oil (McNutt & He, 2016).

Several types of vegetable oils used as raw materials in manufacturing bio-lubricants include castor oil, Pongamia and Simarouba seeds, soybean oil, coconut oil and palm oil (Mobarak et al., 2014; Rios et al., 2020; Tulashie & Kotoka, 2020). Biolubricant performs better than petroleum lubricants, with desirable characteristics such as renewable, low toxicity, and good biodegradable for many applications (Bolina et al., 2021).

Despite the advantages, lubricants are not yet widely used due to several significant challenges and difficulties related to their performance. In addition to concerns regarding the reliability and consistency of raw materials and industrial acceptability. Lubricants also have two significant harmful physical properties for poor low-temperature performance and low thermal-oxidative stability. The modification of plant fatty acid structures has great potential for improving the friction-reducing properties (Salih et al., 2017). However, proper chemical modification can improve these two properties to make lubricants a viable alternative to mineral lubricants for various applications (Ho et al., 2019). Therefore, this study aims to examine the optimum conditions for producing polyol esters and the effect of the temperature of the acetylation on the value of the reaction kinetics constant, as well as the physical and chemical properties of the polyol ester produced from Crude Palm Oil.

METHOD
Materials

Solutions of glacial acetic acid and sodium bicarbonate were purchased from Merck. Bentonite from the supplier was used as the catalyst. As initial compounds to produce polyols, epoxide compounds were obtained by reacting crude palm oil with methanol at 70°C for 90 minutes using 2% H_2SO_4 as a catalyst. The polyol ester was produced from the hydroxylation reaction of the epoxide compound based on Crude Palm Oil and methanol at 45°C for 120 minutes with 1.5%wt bentonites.

Catalyst Activation

Bentonite used as a catalyst was sieved using a 125 mesh sieve. Bentonite was thermally activated at 400°C using a furnace for 6 hours. The bentonite is then cooled and stored in a desiccator.

Synthesis of Polyol Ester Compounds through Acetylation Reaction

The polyol ester compound was synthesized through an acetylation reaction according to the schematic diagram in Figure 1. The acetylation process was carried out by reacting the polyol compound (Table 1) from CPO with 97% acetic anhydrous acid as 10%wt. Bentonite was added to the reaction with variations of 1, 2, and 3%wt. The reaction occurred at various temperatures, 60, 70, and 80 °C, with reaction times of 15, 20, 25, 30, and 35 min. The mixture that has been reacted is then filtered using filter paper to separate the product (liquid) and the rest of the catalyst and reactants. The mixture was separated through a separatory funnel, and then a saturated $NaHCO_3$ solution was added to neutralize 200 mL of acid and distilled water and stirred. The remaining acid was neutralized using a saturated $NaHCO_3$ solution. Neutralization and washing are carried out until it reaches a neutral pH. The compound resulting from the acetylation reaction was allowed to stand for 24 h and then separated. The

oxirane and acid numbers of the analyzed products were determined according to ASTM D1652 and AOAC 1995. Kinematic viscosity and density were analyzed according to ASTM 445 and ASTM D4052.

Product Analysis

The quality of the polyol ester was determined by the oxirane and the acid number of the polyol ester using the ASTM D1652 and AOAC 1995 methods. Meanwhile, the kinematic viscosity and density values were determined based on the ASTM D445 and ASTM D4052 methods. This method was chosen because it meets the standard requirements for the test material. Namely, the test material can be handled generally as a liquid at the test temperature, using manual or automatic sample injection equipment.
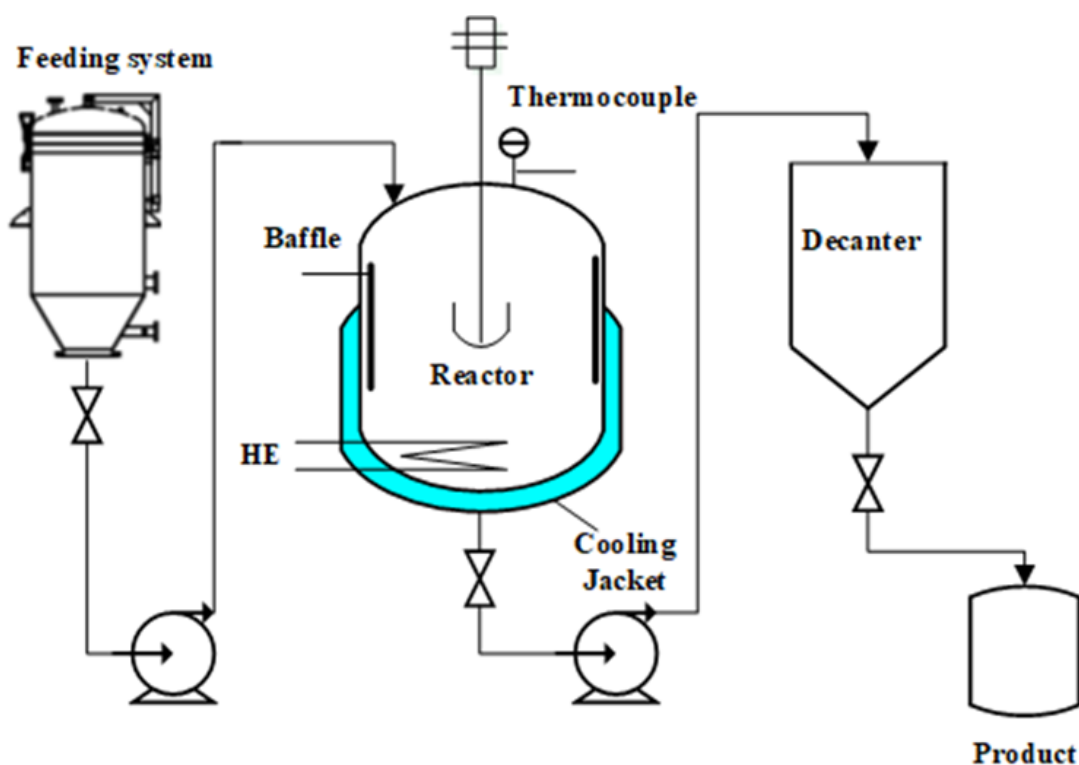
Figure 1. Schematic diagram of the synthesis of polyol ester

Table 1. Analysis of chemical and physical properties of polyol compounds

Physical properties	Unit	Value
Density	g/mL	0.902
Kinematic viscosity	cSt	
40 °C		60.318
100 °C		11.656
Viscosity index		144.305
Acid number	mg NaOH/mg compound	1.552
Oxirane number	%/100 g	3.155

RESULT AND DISCUSSION

Effect of Temperature, Reaction Time, and Catalyst Concentration on the Acid Number of Polyol Ester Compounds

The acid number is closely related to the corrosion rate of the engine due to the use of bio lubricant. The acid number is determined by the amount of base needed to neutralize the acid per one gram of the bio lubricant sample. From Figure 2, the increase in reaction temperature and the length of reaction time can reduce the acid content of the bio lubricant. The number of polyol ester acids produced through the acetylation reaction ranged from 0.1980–0.4080 mg NaOH/g. The highest acid number was obtained at 60°C for 15 min with 1 %wt catalyst, while the lowest acid number was obtained at 80 °C for 35 min with 2 %wt catalyst. The higher the temperature and the longer reaction time will reduce the remaining anhydrous acetic acid, which may not react and cannot be neutralized. Meanwhile, the amount of catalyst concentration did not significantly affect the decrease in acid number. The acid number is kept as low as possible to minimize the risk of engine corrosion.

The Hydroxyl Number of Polyol Ester

The hydroxyl number indicated the number of milligrams of the base to neutralize the acetic acid required for the acetylation reaction of the hydroxyl group per gram of sample. The effect of temperature, reaction time, and catalyst concentration on the hydroxyl number of polyol ester compounds is shown in Figure 3. This hydroxyl number measurement is used to calculate the polyol conversion. From the study results, the hydroxyl number has decreased by increasing the temperature and reaction time. The decrease in hydroxyl number is also influenced by the amount of catalyst used. A higher temperature or catalyst concentration will increase the conversion of the reaction resulting in a decrease in the hydroxyl group in the reaction product. The results also show that temperature and catalyst concentration are interacting factors, so the effect of temperature on the hydroxyl number varies depending on the catalyst concentration (Suárez et al., 2015). The decreased hydroxyl number was due to the conversion of the hydroxyl group into an acetyl group through the acetylation process. The lowest hydroxyl number was obtained at a reaction temperature of 80°C for 35 min and 3%wt catalyst.

In addition to being an indicator of the presence of a converted hydroxyl group, the hydroxyl number is related to the pour point characteristics of the bio lubricant. According to (Chan et al., 2018), the hydroxyl group will give the characteristics of a viscous bio lubricant due to intermolecular hydrogen bonds. At low temperatures, vegetable oils tend to form macro-crystal structures due to the accumulation of triglyceride backs. The crystal structure will hinder the ease of flow due to losing molecular kinetic energy (Salimon et al., 2012). The increase in the hydroxyl group will also increase the number of hydrogen bonds, thus increasing the

pour point. Biolubricant with a high pour point will be difficult to flow in a system that is in a low-temperature environment.

Conversion and Acetylation Reaction Kinetics

The amount of conversion of polyol compounds into polyol ester can be calculated by changing the concentration of polyol compounds before and after the reaction. The conversion of polyol ester is influenced by reaction conditions such as reaction temperature, reaction time, and catalyst concentration (Figure 4). The conversion of polyols to polyol esters increases with variations in reaction time. The highest conversion of polyol ester compounds from CPO (38.41%) was produced at 80 °C for 35 min using 3%wt catalysts. The quantity of reactant molecules in contact increases as the acetylation reaction time increases. The more molecules between reactants in contact, the more reactants react to form products.
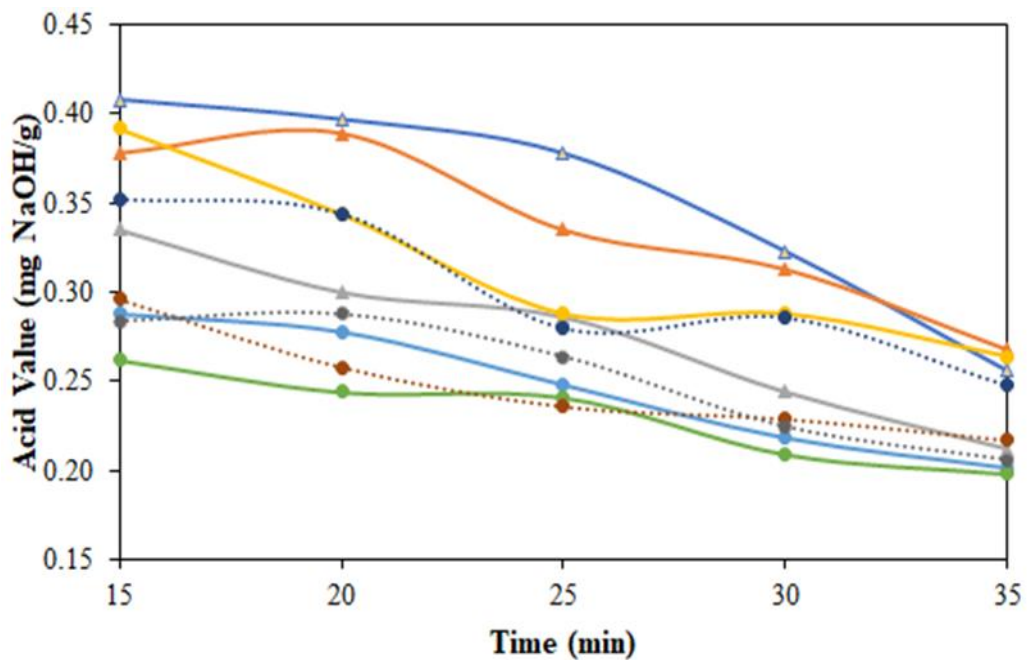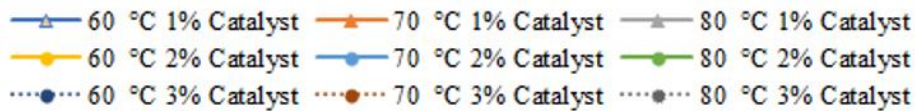

Figure 2. The acid value of polyol ester compounds

—△— 60 °C 1% Catalyst	—▲— 70 °C 1% Catalyst	—▲— 80 °C 1% Catalyst
—●— 60 °C 2% Catalyst	—●— 70 °C 2% Catalyst	—●— 80 °C 2% Catalyst
····●··· 60 °C 3% Catalyst	····●··· 70 °C 3% Catalyst	····●··· 80 °C 3% Catalyst
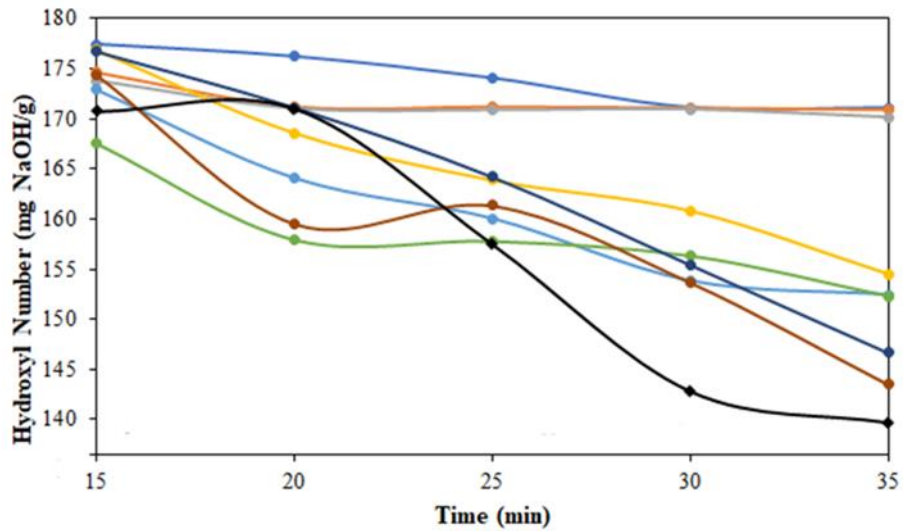
Figure 3. The Hydroxyl Number of Polyol Ester Compounds
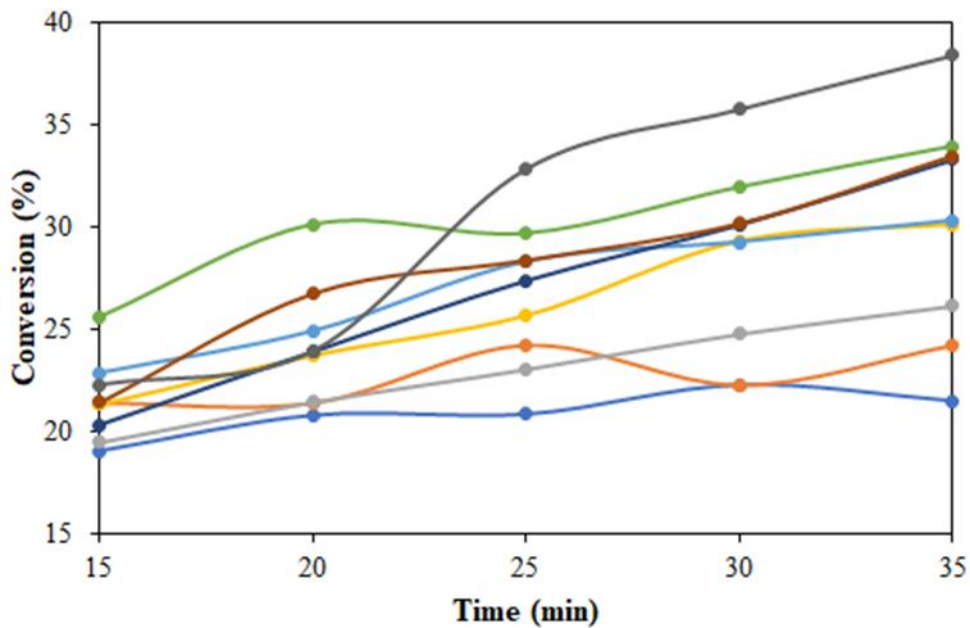
Figure 4. Conversion of Polyol Ester

Bentonite catalyst accelerates the rate of acetylation reaction to reach equilibrium. The ability of a chemical compound to catalyze a reaction can be seen through the activity of the catalyst, which is usually expressed in the percentage of conversion or the number of products produced from the number of reactants used in a specific reaction time (Roni et al., 2021). This trend is caused by an increase in the total number of active sites for the reaction, which can accelerate the reaction rate to reach reaction equilibrium (Ngadi et al., 2015). The increased catalyst concentration resulted in increased conversion. This result is in line with (Arumugam et al., 2019), who stated that the greater the number of catalysts, the greater the conversion and yield. The reaction kinetics is based on the formation rate of the polyol ester compound, which is expressed in the conversion of the polyol ester. The order of the acetylation reaction at each temperature and type of catalyst is pseudo-first order. It is in line with research (Chen et al., 2019). The reaction kinetic constant for each temperature is obtained by using the conversion data. The value of k is obtained from the slope of the linear regression equation graph -ln(1-XA) = k.t versus reaction time (t).

The acetylation reaction constant obtained can be used to calculate the activation energy (E), Arhennius constant (A), and reaction enthalpy (ΔH). The general formula for the value of the reaction constant follows equation 1, which can be simplified again in equation 2.

$$k = Ae^{(-E/RT)} \qquad (1)$$

$$\ln k = \ln A - E/RT \qquad (2)$$

Furthermore, to get the ln A and –E/R value, the relationship graph between ln k and the value of 1/T is made (Figure 5). The graph formed is a graph of a linear equation with intercept and slope values.

Ln A and -E/R values are obtained based on the intercept and slope values. The A value is the resulting intercept, and the -E/R value is the resulting slope. Through equations and calculations, the value of activation energy (E), frequency factor (A), and enthalpy (ΔH) will be obtained for each catalyst concentration and variations in reaction temperature. The value of activation energy (E), frequency factor (A), and enthalpy value (ΔH) for each catalyst concentration, as well as variations in reaction temperature, are shown in Table 2.

The resulting enthalpy value at 80 °C with a catalyst concentration of 3%wt is 14687.10 J/mol. The enthalpy value will decrease with increasing reaction temperature.
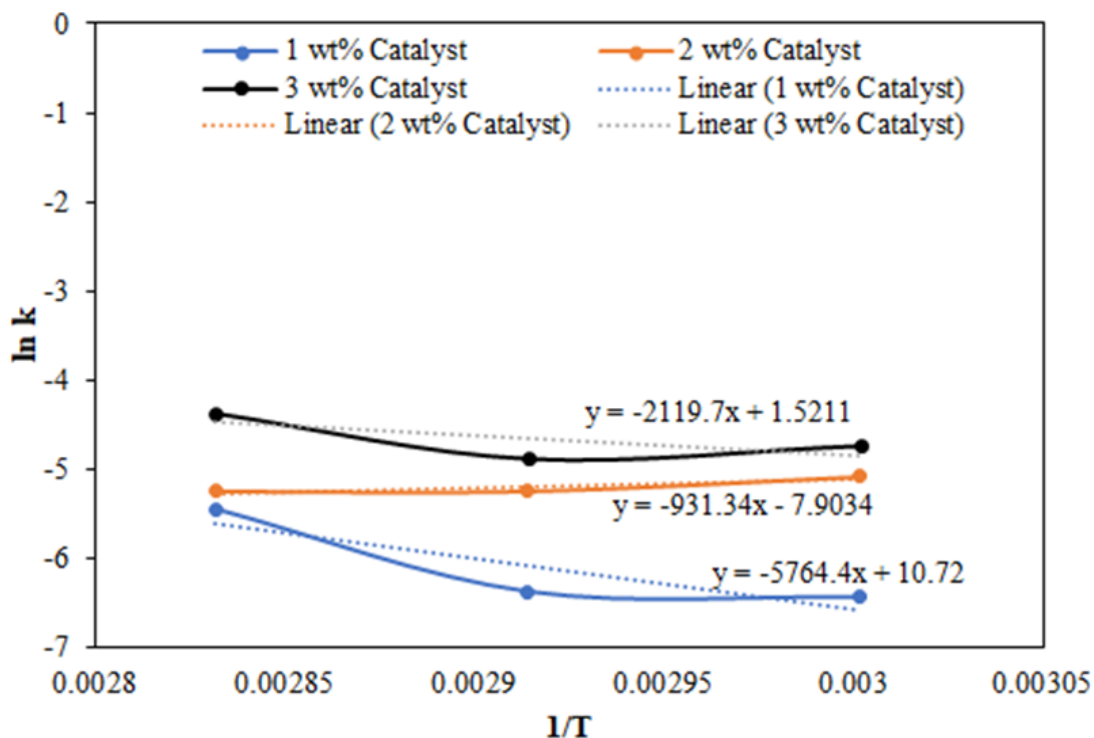
Figure 5. The relationship between ln k and the value of 1/T

Table 2. Value of activation energy (E), frequency factor (A), and enthalpy (ΔH) of acetylation reaction

Catalyst (%wt)	Temperature (°C)	E (J/mol)	A (mol/L.min)	ΔH (J/mol)
	60			45155.41
1	70	47925.2	45251.9	45072.27
	80			44989.13
	60			4991.476
2	70	7743.161	0.000365	4908.336
	80			4825.196
	60			14853.38
3	70	17623.19	4.577257	14770.24
	80			14687.10

Physical and Chemical Properties of Polyol Esters

The acetylation process was carried out by varying the reaction temperature, reaction time, and concentration of catalyst addition. Table 3 shows the results of the polyol ester analysis obtained, including polyol ester density, kinematic viscosity, and viscosity index.

Table 3. Results of analysis of physical and chemical properties of polyol esters produced

Temperature (°C)	Time (min)	Catalyst (%wt)	Density (g/mL)	Kinematic viscosity (cSt)		Viscosity Index
				40 °C	100 °C	
60	15	1	0.9468	45.6511	11.6556	165.5257
		2	0.9231	47.6908	14.4724	169.0300
		3	0.9361	48.0794	14.5695	168.8862
	20	1	0.9330	45.8213	12.5138	167.5944
		2	0.9388	46.8167	12.6269	166.7793
		3	0.9230	45.8811	12.6085	167.6823
	25	1	0.9440	47.2052	12.9183	167.0606
		2	0.9411	48.0794	15.3465	169.3852
		3	0.9178	43.7085	15.5408	172.2062
	30	1	0.9430	45.1355	11.5613	165.8271
		2	0.9123	45.6511	11.4613	164.8652
		3	0.9330	45.8754	11.6658	165.2742
	35	1	0.9526	46.4281	11.6556	164.6546
		2	0.9385	43.9999	10.6843	163.7098
		3	0.9435	45.5540	10.4900	160.5667
70	15	1	0.9342	43.7085	10.8786	164.9735
		2	0.9261	46.2339	9.7130	154.3089
		3	0.9352	48.5650	10.5872	157.0127
	20	1	0.9530	46.6114	11.5874	164.1422
		2	0.9498	47.5937	11.6556	163.3479
		3	0.9530	46.2283	11.6920	164.8786
	25	1	0.9183	43.7085	14.0839	171.7192
		2	0.9297	43.1257	15.1523	172.5957
		3	0.9213	44.6798	15.3465	171.6101
	30	1	0.9430	47.0537	13.6744	168.6348
		2	0.9543	48.5650	13.5982	167.2475
		3	0.9430	47.5899	13.5671	168.0547
	35	1	0.9200	47.3023	18.4547	169.8699
		2	0.9487	45.1655	11.4613	165.4292
		3	0.9622	44.6798	15.5408	171.5871
80	15	1	0.9616	42.7372	11.6556	168.7924
		2	0.9215	42.0573	11.2671	168.4623
		3	0.9447	41.8630	11.9470	170.2459
	20	1	0.9530	44.5133	11.1105	164.7994
		2	0.9186	45.4568	11.0728	163.6222
		3	0.9230	45.6874	11.2610	164.0955
	25	1	0.9343	40.7946	13.8896	173.9139
		2	0.9251	43.7085	17.4834	171.9081
		3	0.8846	44.5827	15.5408	171.6490
	30	1	0.9130	45.2265	12.8047	168.6668
		2	0.9037	47.5937	12.7240	166.3558
		3	0.9330	46.1578	12.1128	166.2916
	35	1	0.9005	44.2913	11.6556	167.0501
		2	0.9005	45.3597	9.8101	156.3951
		3	0.9147	46.2339	8.7417	144.0818

From the density measurement results of the resulting polyol ester, the density varies from 0.885-0.962 g/cm3. Biolubricant does not have a specific limit for the density value, but the density of conventional lubricants generally varies from 0.860 to 0.980 g/cm3 (Rashmi et al., 2017). Compared with the density of petroleum lubricants analyzed by (Egbuna et al., 2021), the polyol esters produced have a relatively high-density range compared to petroleum lubricants, namely 0.848. It can be caused by changes in the chemical structure of the constituent molecules. It should be noted that a change in specific gravity leads to a corresponding change in the mass of the product. In other words, the higher the specific gravity, the heavier and thicker the lubricating oil. Therefore, the bio lubricant has a better advantage at high temperatures in terms of resistance and the ability to withstand greater loads. Their density determines the compatibility of bio lubricant products with heavy or light working machines. Compatibility is the ability of a sample to mix with other liquids. Thus, materials with lower density (<1) float in water, while materials with higher density (>1) sink in water. Therefore, lubricants with higher density and viscosity last longer on the surfaces and joints to which they are applied (Sapawe et al., 2019; Yang et al., 2020).

Viscosity index is defined as a liquid's property that helps resist changes in viscosity with an increase or decrease in temperature. In lubricants, it is essential to have a higher viscosity index value. It is because lubricants with a higher viscosity index can significantly withstand changes in viscosity with temperature changes. The viscosity index obtained is in the range of 144.0818 – 173.9139. It value is greater than the one of petroleum lubricants. A high viscosity index is characteristic of compounds with long, unbranched hydrocarbon chains like the fatty acids of most natural triglycerides (Encinar et al., 2020; Moreira et al., 2020). Adding viscosity stability enhancing additives can increase the viscosity index value based on the desired value (Ahmed et al., 2014; Shara et al., 2018). In addition to density, kinematic viscosity, and viscosity index, several physical and chemical properties of polyol ester compounds produced from CPO with the highest conversion at 80 °C for 35 min using 3%wt catalysts have also been analyzed, which are summarized in Table 4.

Table 4. Physical and Chemical Properties of Polyol Ester

Analysis	Unit	Method	Value
Density at 20 °C	g/cm^3	ASTMD-1298	0.915
Kinematic viscosity at 40 °C	cSt	ASTM D445	46.2339
Kinematic viscosity at 100 °C	cSt	ASTM D445	8.7417
Viscosity index	-	ASTM D2270	144.0818
Flash point COC	°C	Flash point Kohler; GB/T3536-2008 & ASTMD 92	282.3
Pour Point	°C	ASTM D97	45
Total base number	mg KOH/g	ASTM D974	0

CONCLUSION

Production of bio lubricant based on crude palm oil through acetylation reaction has been carried out. This study aims to determine the characteristics of the bio lubricant produced. The most extensive conversion of polyol esters was 38.41% at 80°C for 35 min using a 3%wt bentonite. Operating conditions affect the resulting % conversion. The higher the reaction temperature, the length of reaction time, and the concentration of the catalyst used, the greater the conversion produced.

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